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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: FIFE, *et al.*

Application No.: 09/632,714

Filed: August 4, 2000

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Group Art Unit: 1775

Examiner: S. STEIN

For: NIOBIUM POWDERS AND NIOBIUM ELECTROLYTIC CAPACITORS

**DECLARATION OF JONATHON L. KIMMEL**Assistant Commissioner for Patents  
Washington, D.C. 20231

May 21, 2001

Sir:

I, Jonathon L. Kimmel, declare and state as follows:

1. I am currently an employee of Cabot Corporation, the Assignee of the above-identified patent application. Furthermore, my responsibilities at Cabot Corporation include being Manager of the research and development of niobium based products including the invention set forth in the above-identified patent application. I have been employed by Cabot Corporation since February 1, 1998. Furthermore, my education background includes holding a Bachelor's Degree in electrical engineering and doing prior work with such companies as DuPont Corporation.

2. I am familiar with the invention and the claims set forth in the above-identified patent application as well as the Office Action dated January 19, 2001. Furthermore, I am familiar with the documents referenced by the Examiner including Chang, Schiele, and Fry.

3. From the Office Action, it is my understanding that the Examiner is arguing that tantalum and niobium are similar such that substitution of each powder is possible.

4. In response to the Examiner's comments set forth in the Office Action, it is my opinion, which is based on experience and knowledge in this area, that niobium can not simply be substituted for tantalum especially in the electrode capacitor area.

Declaration of Jonathon L. Kimmell

5. To further support this point, attached is information from the Tantalum-Niobium International Study Center (also known as TIC) that further explains the current applications for tantalum and niobium. While all the applications may not be mentioned, it is important to realize that the first application mentioned for tantalum is capacitors. However, there is no mention under the niobium section for capacitors. This further confirms that niobium and tantalum are not easily substituted contrary to the Examiner's reasoning. In addition, at page 3 of the TIC printout, there is a history of tantalum and niobium identified as "Tantalum And Niobium-Dissimilar Twins?" While this section does not fully explain the differences between tantalum and niobium, it is interesting to note that this brief article appreciates the significant differences between tantalum and niobium.

6. In addition, attached are graphs related to worldwide capacitor consumption which are from the TIC and are publicly available. As can be seen, from the bar graphs, the production of niobium was so insignificant for capacitor consumption that it did not even record on the bar chart for 1990. This is even true for 2000. Similarly, in the table that shows worldwide capacitor consumption for 1995 and 2000, again, it can clearly be seen that the consumption for niobium was non-existent. Thus, it is clear that niobium even as of 1990 and as of the filing date of the present application was not used commercially in capacitor manufacturing. Thus, if niobium was an easy substitute for tantalum, manufacturers would be using niobium because it is less expensive and is more available. However, manufacturers are not using niobium in the capacitor market. It is clear that niobium and tantalum are different and niobium is not a substitute for tantalum capacitors. One reason is because the pentoxide layer formed on the niobium anode readily becomes unstable for the life of the capacitor thus resulting in the pentoxide film becoming less stable and causing a breakdown of the capacitor. This has been shown by accelerated life testing of the previous niobium

Declaration of Jonathon L. Kimmell

capacitor's problem. This problem does not occur in tantalum capacitors. To overcome this problem, the present invention uses high surface area niobium and preferably uses various dopants such as oxygen and the like.

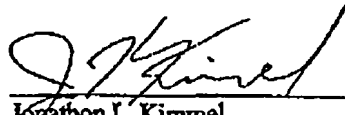
7. In addition, as set forth in the beginning of the present application, niobium has never been considered as a substitute for tantalum by any commercial industry or manufacturer of the powders. This is, as stated above, confirmed by the charts that show capacitor consumption which has not included niobium. Furthermore, according to the Examiner, if tantalum and niobium are so similar, why is the DC leakage different at formation voltages above 60 volts? This is confirmed by the attached charts comparing niobium with tantalum with respect to various formation voltages in DC leakage. As can be seen, the DC leakage for niobium rapidly increases at formation voltages above 50 volts which is quite different from tantalum powders. In addition, the capacitance capabilities of niobium versus tantalum are quite different as well as the effect of sintering temperatures for DC leakage. This is reflected in the attached charts which show these differences with respect to capacitance based on formation voltages.

8. Thus, as indicated by the various evidence set forth above, niobium is a separate product from tantalum and niobium has different properties from tantalum with respect to the capacitance achieved, the DC leakage, the crush strength, and other properties related to using niobium in the formation of an anode when compared to tantalum being used in the formation of an anode. Thus, there is not simple interchangeability of niobium for tantalum.

**Declaration of Jonathon L. Kimmell**

I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

5/21/07  
Date

  
Jonathon L. Kimmell



## The metals

### TANTALUM



Some current applications:

1. As metal powder for the production of tantalum capacitors (dependent on the insulating property of the tantalum oxide films) and as metal wire for their connection to circuits. This biggest single use depends on the large surface area of the finely divided powder, and on the purity of the metal.
2. As fabricated metal for the construction of chemical process plant, and equipment such as heat exchangers, due to its resistance to corrosion.
3. As an ingredient of superalloys, principally for use in aircraft engines and spacecraft.
4. As the carbide, in hard metal cutting tools, also with tungsten carbide and cobalt binders.

The first item accounts for about 50% of current tantalum usage.

### NIOBIUM



Some current applications:

1. In structural steels (especially high strength low alloy (HSLA) steels with up to 1% Nb). In heat resisting steels and superalloys (iron, nickel or cobalt based).
2. As an alloy with titanium or tin for superconductive magnets.
3. As the oxide ( $\text{Nb}_2\text{O}_5$ ) in high refractive index glass, as lead niobate in piezoelectric devices, and as single crystal lithium niobate in surface acoustic wave filters for television sets and similar equipment.
4. With copper in powder metallurgy composites for components requiring high strength with conductivity.
5. As an alloying addition to zirconium for nuclear reactor fuel tubes.

6. As the carbide, in cutting tools, with tungsten and tantalum carbides.
7. As the fabricated metal, for chemical plant, and, alloyed with zirconium or other metals, for high temperature applications in aerospace.

The first item accounts for about 90% of current niobium usage.

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### The association: T.I.C.

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Tantalum-Niobium International Study Center,  
40 rue Washington,  
1050 Brussels,  
Belgium.  
Telephone: +32 2 649 51 58  
Fax: +32 2 649 64 47  
e-mail: [tanlniob@agoranet.be](mailto:tanlniob@agoranet.be)

The Tantalum-Niobium International Study Center is an international, non-profit association founded under Belgian law: it received Royal Assent in 1974, and has its headquarters in Brussels, Belgium. It has more than 50 members, which are companies involved in raw material production, processing, assaying, trading, capacitor manufacture, chemical process equipment manufacture, and are situated in more than 20 different countries across the world. A list of the current members is included in this site, with an indication of the main field of activity of each. The association is managed by an Executive Committee with members drawn from all parts of the industry and regions of the world.

#### Alms

The main goal of the T.I.C. is to increase awareness of tantalum and niobium and their remarkable properties, and to promote the use of the metals and their products. The association organises each October a conference with technical and business presentations, and the programme also includes a plant tour to a factory or mine associated with the production or use of these metals, sponsored by one of the members. At intervals the T.I.C. also organises an international Symposium of about three days, and such a meeting is planned for the west coast of the United States in October 2000. A quarterly Bulletin with some of the presented papers and other articles of general interest to the industry is published: copies may be requested from the Secretariat. The association also collects statistics on the industry, these are supplied to the members, who also receive a monthly set of abstracts and news headlines.

#### Membership

Companies which have a continuing interest in one or both of the metals are eligible to apply for membership. Information and application forms may be obtained from the Secretariat.

Associate membership is available to individuals and to non-profit organisations with an interest in the metals which is not commercial, such as researchers, academics, authors.

### Tantalum and Niobium — Dissimilar Twins?

In 1801 an American chemist named Hatchett was testing a heavy black mineral from Connecticut and discovered that it contained a new element which he named 'Columbium'. A year later Eckberg in Sweden discovered two minerals each containing an oxide of an unknown element. This proved very difficult to dissolve in acids and frustrating to work with, so he named it 'Tantalum' after the Greek god Tantalus (who could not reach the water to drink or the apple to eat).

In 1844 the chemist Rose showed that another element was present in the Swedish mineral, and he named it 'Niobium' after Niobe, the daughter of Tantalus. Only in 1866 did Marignac develop a method of separating the two elements chemically by taking advantage of the difference in solubility of the two potassium double fluorides (a procedure used until quite recently in the manufacture of the metals). The European 'Niobium' was soon shown to be identical to the American 'Columbium' and for nearly a century arguments raged over which name had priority. Finally in 1950 the international chemical body, by a majority decision, settled for niobium but the old name columbium is still in common use in the Americas, complicated by the fact that one of the two most common minerals of niobium is universally known as 'Columbite'. Of the two elements, niobium is far more abundant in the earth's crust than tantalum; nevertheless they almost always occur together. This results from the great chemical similarity of their oxides (which gave those early chemists so much trouble), and from their very similar atomic radii, so that they freely replace each other in minerals. The columbite mentioned above is an iron or manganese niobate and there is an iron manganese tantalate known as tantalite. A full range of mixtures between the extremes exists, all naturally occurring.

When the two elements were finally separated, and the metals produced, it was obvious that the similarity did not extend to all their physical properties. Niobium metal is very similar in density to iron, but tantalum is nearly twice as heavy. As a result of all these factors, and the relative abundance (and cheapness) of niobium, they have very different applications, but in some uses, in particular of the high purity metal and its alloys, there is some overlap. Dissimilar twins indeed!

Applications of tantalum and niobium are based on their ability to form a non-conducting layer of oxide on the surface of the metal, a dense, stable and adhesive layer of pentoxide.

#### Tantalum — Raw Materials and Processing

Until recently the majority of the world's production of tantalum was from the discard slags of tin smelters. The tin mineral cassiterite is frequently associated with columbite/tantalite ore, especially in Thailand, Australia, Brazil and Central Africa, and to a smaller extent in Malaysia. When the tin concentrates are smelted, the tin is reduced to metal, but the tantalum reports unreduced in the slag, from which it can be recovered by electric smelting and/or chemical extraction. Tin slags, particularly those from the Thaisarco smelter in Phuket, Thailand, used to be an important supplier of tantalum, but the decline of the tin industry in South East Asia since 1985 has meant the replacement of much of that source of tantalum by primary

#### Niobium — Raw Materials and Processing

A small part of industry's needs for niobium is recovered during the treatment of tin slags and columbite/tantalite minerals (mined in West Africa, Brazil, South East Asia and Australia) which are the source of much of the world's tantalum. However, more than 90% of all niobium is recovered as ferroniobium for use in steel-making, from the smelting (by reduction with aluminium) of the mineral pyrochlore, a calcium fluoniobate (which, when the niobium is replaced by tantalum, is known as microlite, and mined for tantalum). Two mines in Brazil (the CBMM mine at Araxá, and the Anglo-American mine at Catalão in Goiás) account for 80% of all niobium, and one in Canada (Niobec at St Honoré) for more than half the remainder.

mining of tantalite.

There are a number of mines now operating, two of the largest are open-cut operations in Western Australia (Greenbushes and Wodgina). A minor source resulted from a boom in tantalum prices in 1979/80 which caused the excavation of very large tonnages of old tantalum-bearing tin slags in South East Asia, where they had been used as ground fill since early this century. Some of these are still supplying the processors' needs but it is generally considered that stocks of them are now small. There is also a sizeable amount (about 25%) of recycling of scrap metal and of compounds of tantalum.

Tantalum and niobium are extracted from their ores, after concentration, by chemical means rather than by smelting. The concentrates are attacked by  $\text{HF}/\text{H}_2\text{SO}_4$  which brings the tantalum and niobium compounds into solution. The acid solution is mixed thoroughly with MIBK (methyl-iso-butyl ketone) which dissolves the tantalum and niobium compounds into the ketone while leaving impurities in the aqueous solution. The organic and inorganic solutions form separate layers and the organic (ketone) solution can be separated from the aqueous layer (liquid-liquid separation). The niobium is then stripped with dilute acid, and the tantalum subsequently extracted by acid ammonium fluoride. For tantalum, the metal is usually produced in powder form by sodium reduction of the fluoride. It can then be compacted (as it is for capacitors) to final shape, or may be melted (and refined) in an electron beam furnace.

Pure niobium and its oxide are prepared through a chemical route similar to that applied to tantalum.



#### Tantalum — Applications

For a brief period early this century, tantalum had a use in wire form as lamp filament, but the development of cheap tungsten wire put paid to this.

The biggest single use now of tantalum (accounting for 50% of the total) is as the powdered metal, mostly for the production of capacitors. These depend on the dielectric properties of a tantalum oxide film on a tantalum compact, but the amount of tantalum required for a particular service is reduced as the metal purity is raised. Technical advances in tantalum manufacture and increasing knowledge of the effect of other parameters such as particle size and shape have enabled capacitor makers to make large increases in their production of units with little significant

#### Niobium — Applications

The ferro-niobium which makes up most of niobium consumption is used in the preparation of steels, in particular those of the HSLA (high strength low alloy) type. Until recently the best-known use of the niobium-bearing steel has been in the manufacture of natural gas pipelines: the niobium gives the steel the strength to resist the great range of temperatures to which such pipes are subject. Niobium addition is now growing in other steels such as stainless, and this has been helped by the stability of its price compared with that of competing alloying elements.

Pure niobium, like tantalum, has considerable attraction as a material of construction for chemical plant, and in the formulation of superalloys, but it has an additional use which

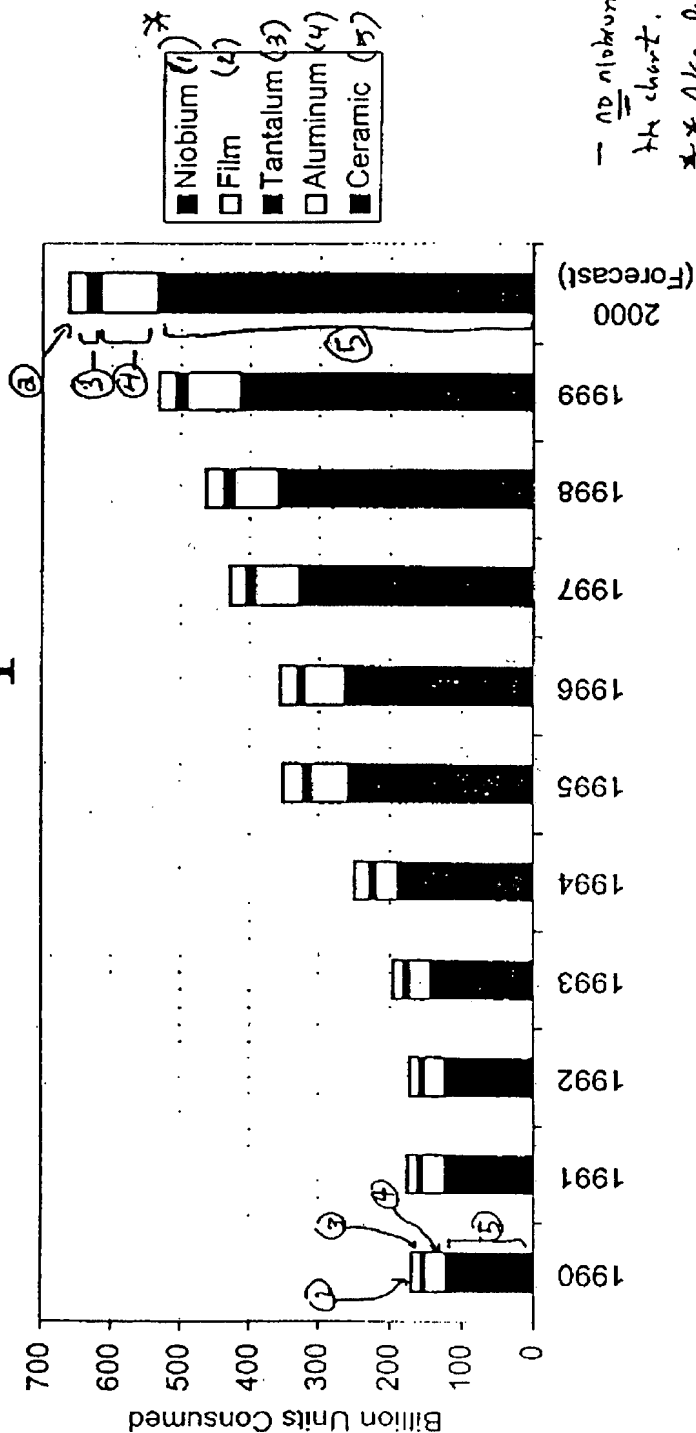
increase in the weight of tantalum consumed. Also important as applications of tantalum are tantalum carbide used in cutting tools (with titanium and tungsten carbides), and pure or alloyed tantalum metal, much of it for corrosion or heat resistant chemical plant equipment, or in superalloys for jet engines



alloyed with titanium or tin, as a superconductor. Superconducting applications include magnetic resonance imaging for medical diagnostics and in particle accelerators for physics research. The Mag-Lev train may be a more conspicuous use of this material in the future. The high strength magnetic fields required can be generated in a small volume by superconducting coils — in medicine, the technique is much safer than that using X-rays.

Niobium also has several non-metallic applications. An important one is in optical glass: when the silica in glass is replaced by niobium oxide, the refractive index is much greater, so thinner and lighter lenses can be used for the same focal length. This is already in use in many camera lenses and for eyeglasses.

# Worldwide Capacitor Consumption



no niobium made  
the chart.  
\* Also, for all other  
bars - the same  
order is followed.

Sources: Tantalum-Niobium International Study Center (TIC);  
Paumonok Publications; Business Process Engineering(BPR)

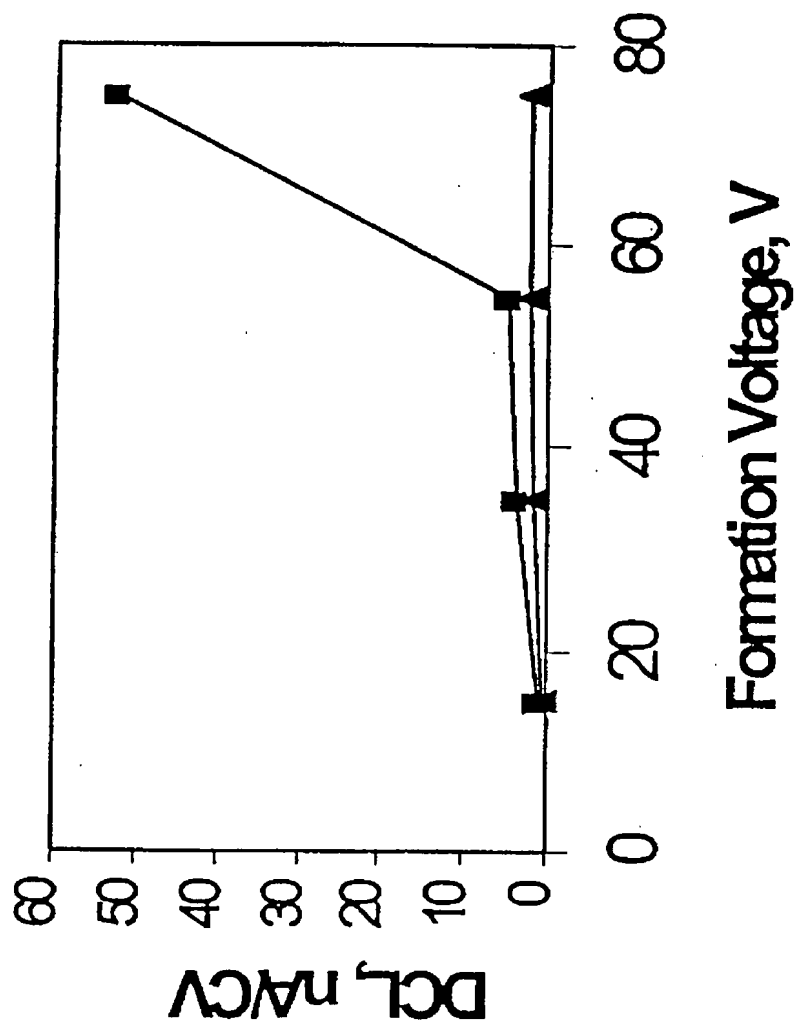
# Worldwide Capacitor Consumption

	1995	2000
WW Consumption (Billion Units)		
MLCC	180	350
SLC	65	80
Aluminum	80	120
DC Film	28	34
Tantalum	14	25
Other	1	1
<b>Total</b>	<b>368</b>	<b>610</b>

Source: Tantalum-Niobium International Study Center (TIC)  
as reported in Electronic Buyers' News 25-Jan-99, p36

# DCL vs Formation Voltage

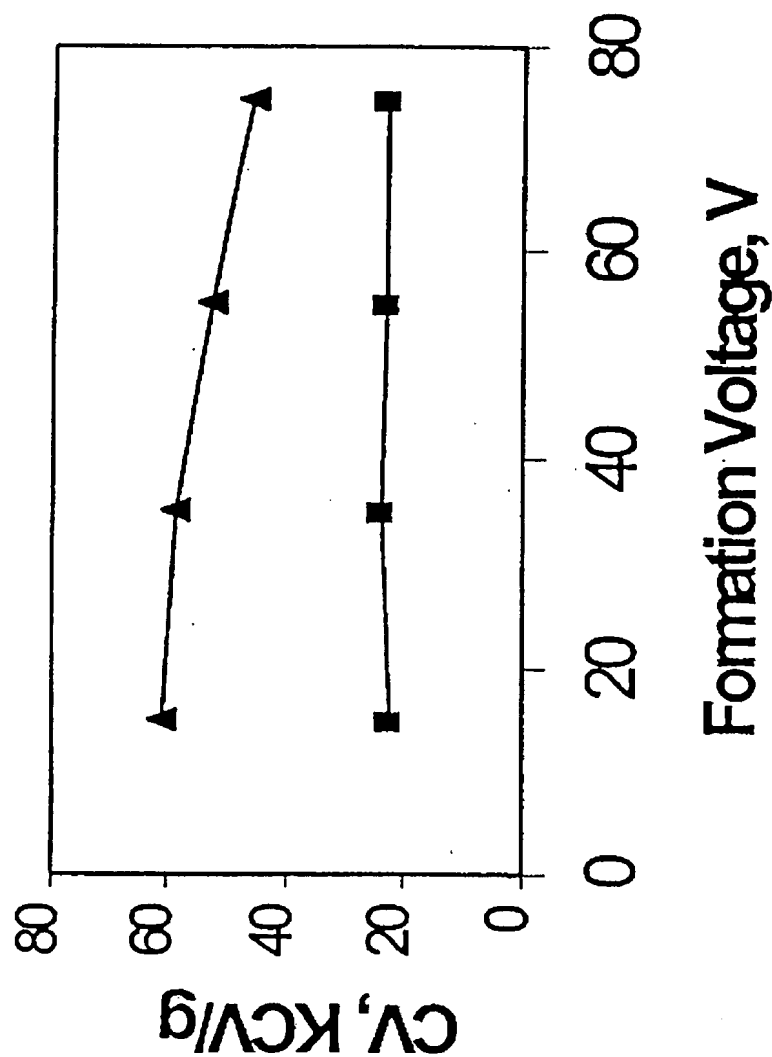
1300 Deg Sinter



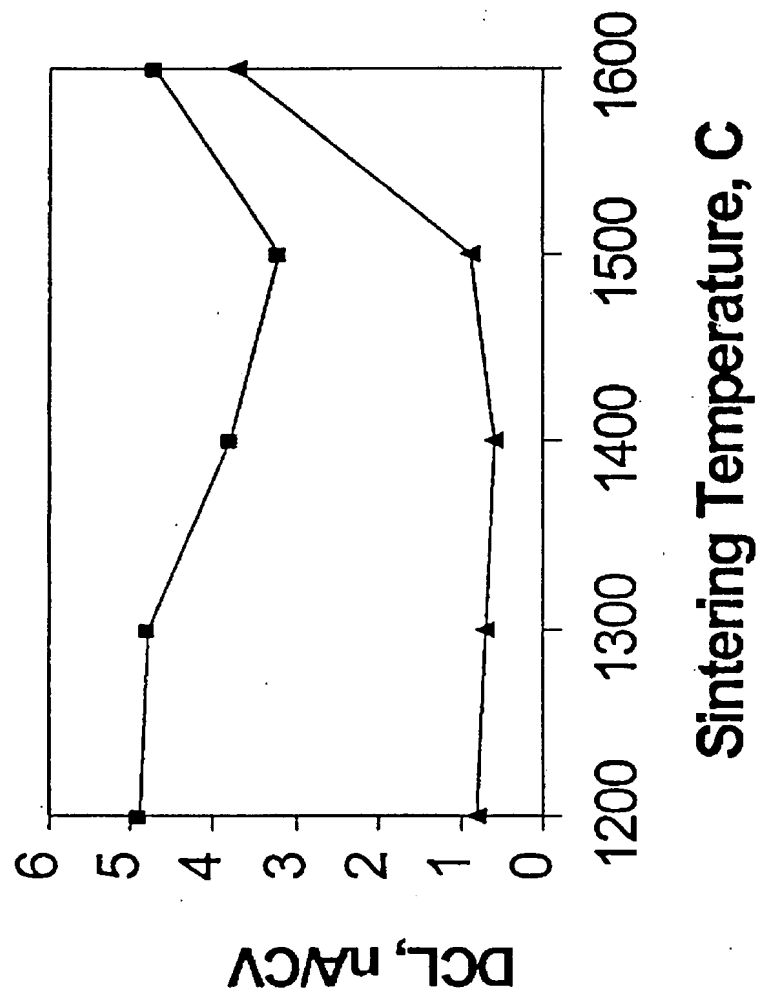
—■— Nb —▲— Ta-C606

# CV vs Formation Voltage

1300 Deg. Sinter

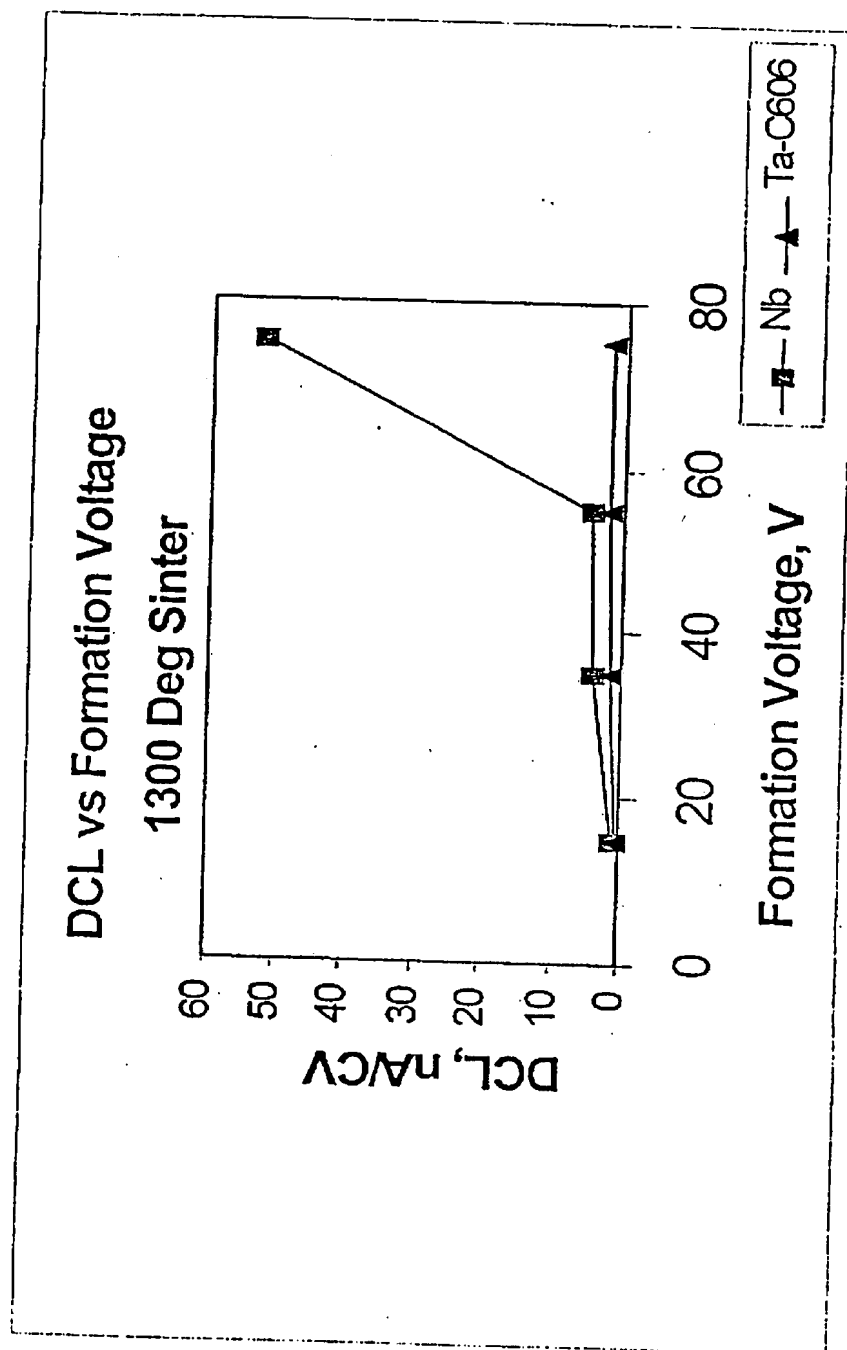


# DCL vs Sintering Temperature



—■— Nb —▲— Ta-C606

## Niobium Vs. Tantalum Forming Voltage Range



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Experiment

Experiment:

Process\*

Product\*

Product Category\*

7007-R51000

Performance Characteristics\*

Responsibility: Dan White

Date: 03/21/2000

Title:

Exp# 2452 (c) Nb vs Ta C606 Anodization Voltage/85C Evaluation

Objectives:Procedures:[1] Samples:

- (a) 8171-33-C 1300 sintered anodes from Exp# 2452(a)
- (b) C606-C-144577 1300 sintered anodes from Exp# 2452(a)

[2] 15V Ef Anodization:

- (1) One Formation
  - N = 8 anodes per sample
  - (3) samples \* (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H<sub>3</sub>PO<sub>4</sub> @ 85 C Deg 4.3mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 15.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[3] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing --
  - 70% Ef Test Voltage
  - 60 and 120 second charge time
  - 10% H<sub>3</sub>PO<sub>4</sub> @ 21 Deg C
- (b) Capacitance-DF Testing:
  - 18% H<sub>2</sub>SO<sub>4</sub> @ 21 Deg C
  - 120 Hz

[4] 35V Ef Anodization:

- (1) One Formation
  - N = 8 anodes per sample
  - (3) samples \* (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H<sub>3</sub>PO<sub>4</sub> @ 85 C Deg, 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 35.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[5] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing --
  - 70% Ef Test Voltage
  - 60 and 120 second charge time

10% H<sub>3</sub>PO<sub>4</sub> @ 21 Deg C  
(b) Capacitance-DF Testing:  
18% H<sub>2</sub>SO<sub>4</sub> @ 21 Deg C  
120 Hz

**[6] 55V Ef Anodization:**

- (1) One Formation  
N = 8 anodes per sample  
(3) samples \* (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H<sub>3</sub>PO<sub>4</sub> @ 85 C Deg 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 55.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

**[7] DC Leakage/Capacitance-ESR Testing:**

- (a) DC Leakage Testing ---  
70% Ef Test Voltage  
60 and 120 second charge time  
10% H<sub>3</sub>PO<sub>4</sub> @ 21 Deg C
- (b) Capacitance-DF Testing:  
18% H<sub>2</sub>SO<sub>4</sub> @ 21 Deg C  
120 Hz

**[8] 75V Ef Anodization:**

- (1) One Formation  
N = 8 anodes per sample  
(3) samples \* (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H<sub>3</sub>PO<sub>4</sub> @ 85 C Deg 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 75.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

**[9] DC Leakage/Capacitance-ESR Testing:**

- (a) DC Leakage Testing ---  
70% Ef Test Voltage  
60 and 120 second charge time  
10% H<sub>3</sub>PO<sub>4</sub> @ 21 Deg C
- (b) Capacitance-DF Testing:  
18% H<sub>2</sub>SO<sub>4</sub> @ 21 Deg C  
120 Hz

**[10] 100V Ef Anodization (After review of 75V Evaluation):**

- (1) One Formation  
N = 8 anodes per sample  
(3) samples \* (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H<sub>3</sub>PO<sub>4</sub> @ 85 C Deg 4.3mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 100.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

**[11] DC Leakage/Capacitance-ESR Testing:**

**(a) DC Leakage Testing --**

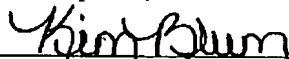
70% Ef Test Voltage  
60 and 120 second charge time  
10% H3PO4 @ 21 Deg C

**(b) Capacitance-DF Testing:**

18% H2SO4 @ 21 Deg C  
120 Hz

I hereby certify that this correspondence is being facsimile transmitted to the United States Patent and Trademark Office, Fax No. 1-571-273-8300 on April 30, 2007.

Kim Blum  
Name (Print)

  
Signature

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	HABECKER et al.	)	Examiner:	Kathleen A. McNelis
Application No.:	10/795,968	)	Group Art Unit:	1742
Filed:	March 8, 2004	)	Confirmation No.:	8631
Docket No.:	99066CON2 (3600-198-02)	)		

For: HIGH CAPACITANCE NIOBIUM POWDERS AND ELECTROLYTIC CAPACITOR ANODES

DECLARATION UNDER 37 C.F.R. § 1.132

Mail Stop Amendment  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

April 30, 2007

Sir:

I, Heather Enman, do declare and state as follows:

(1) I am currently employed with Cabot Corporation, and have been working in the niobium powder area, doing research and product development since 2001.

(2) I have a bachelor's degree and a master's degree in Chemical Engineering from Rutgers University.

(3) I am familiar with the Office Actions dated September 26, 2005 and October 30, 2006, received in the examination of U.S. Patent Application No. 10/795,968. I am also familiar with the cited references relied upon by the Examiner.

(4) In my previous Declaration dated February 24, 2006, the Examiner's position was that the tantalum powder of Chang could be niobium powder and, therefore, in my previous Declaration, it was shown that niobium powders having a BET around 0.55 m<sup>2</sup>/g would not have

U.S. Patent Application No. 10/795,968  
Declaration Under 37 C.F.R. § 1.132  
Reply to Office Action of October 30, 2006

the capacitance set forth in claim 36 of the present application.

(5) From the most recent Office Action dated October 30, 2006, it is my understanding the Examiner requested data from the standpoint of a formation voltage of 20 volts. As can be seen by the attached data, Table 1, a Nb powder having a BET of  $0.58 \text{ m}^2/\text{g}$  when formed at a formation voltage of 35 volts, a formation temperature of  $60^\circ \text{C}$ , and at a sinter temperature of  $1,300^\circ \text{C}$ , for a sinter time of 10 minutes, had a capacitance of 26,286 CV/g. Using the data points in the attached Table, as shown on the attached graph, the affects of sintering temperature can then be extrapolated to show that a capacitance of 26,286 CV/g can be extrapolated to a capacitance of about 40,000 CV/g for a sinter temperature of  $1,100^\circ$ . Further, the same lot of niobium powder was tested at a formation voltage of 30 volts and 35 volts, as shown on the attached Table. From knowing the effects of various formation voltages for this powder, it was possible to determine a mathematical relationship such that a prediction could be made as to what the capacitance would be at a formation voltage of 20 volts, as shown on the attached Table and Figure. In addition, from this prediction, as was done in my earlier Declaration, a prediction was then made on what the capacitance would be at a formation voltage of 20 volts and a sintering temperature of  $1100^\circ \text{C}$ . In making this graph, I have assumed a linear relationship, which is a reasonable assumption, and the resulting capacitance for the  $0.58 \text{ m}^2/\text{g}$  BET sample would be about 43,600 CV/g at  $1,100^\circ \text{C}$  and a  $V_f$  of 20 volts. This is significantly below the 65,000 CV/g stated in claim 36. Even if a fair degree of non-linearity exists in the CV/g—sintered temperature relationship, the capacitance for the  $0.58 \text{ m}^2/\text{g}$  BET sample would still be below 65,000 CV/g in my opinion based on my work in the niobium powder area.

(6) Furthermore, if the tantalum powder of Chang, which has a BET of less than  $0.6 \text{ m}^2/\text{g}$ , was tested for electrical properties at a temperature of  $1100^\circ \text{C}$  for 10 minutes and anodized using a formation voltage of 20 volts at  $60^\circ \text{C}$ , it is extremely unlikely that any anode would form in

U.S. Patent Application No. 10/795,968  
Declaration Under 37 C.F.R. § 1.132  
Reply to Office Action of October 30, 2006

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order to determine the electrical properties of the tantalum powder due to the low surface area of the tantalum powder and, moreover, the DC leakage of such an anode, even if it was formed, would be greatly above 5.0 nA/CV. This is due to the low surface area of the tantalum and that tantalum is not the same as niobium, even when the surface areas are similar. A tantalum powder run at these test conditions would essentially be considered a near-failure or failure.

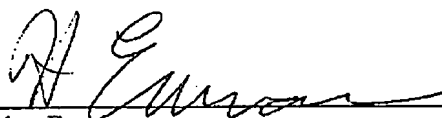
(7) Accordingly, it is in my opinion that the powders of Chang, with respect to niobium powders, would not be capable of the electrical characteristics set forth on claim 36 of the present application.

(8) I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date

4/30/07

Heather Enman



Attachment I: Experiments -- Graph

## Niobium Metal Samples

Sample ID	BET (m <sup>2</sup> /g)	Press Density (g/cc)	Formation Voltage	Formation Temp (C)	Sinter Temp (C)	Sinter Time (min)	CV/g	nA/CV
8656-85-E	0.58	3.0	25	60	1300	10	26463	0.51
8656-85-E	0.58	3.0	30	60	1300	10	26452	0.43
8656-85-E	0.58	3.0	35	60	1300	10	26286	0.78
8656-85-E Predicted	0.58	3.0	20	60	1300	10	26577	
Predicted from cap slope with temperature for 20Vf								
8656-85-E	0.58	3.0	20	60	1100	10	43619	
8656-85-E	0.58	3.0	35	60	1100	10	39296	

CV/g at 20Vf and 1300C sinter is predicted by the equation  $y = -17.7(x) + 26931$

CV/g at 35Vf and 1100C sinter is predicted by the equation  $y = -65.05(x) + 110851$

Cap change from 35Vf to 20Vf is an increase of approximately 11%, therefore CV/g at 20Vf and 1100C for this powder is

$$CV/g = 43619 = 39296(0.11) + 39296$$

Formation

Current Density 50mA/g

35Vf @ 60 Deg C./0.1% H3PO4 Electrolyte

DC Leakage Testing

70% Ef (24.5 VDC) Test Voltage

80 second charge time

10% H3PO4 @ 21 Deg C.

Capacitance Testing:

18% H2SO4 @ 21 Deg C.

120Hz

